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# (54) Method and device for the production of carbon nanotubes

Verfahren und Vorrichtung zur Erzeugung von Kohlenstoffnanoröhren

Procédé et dispositif pour la production de nanotubes de carbone

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## Description

This invention relates to a method of producing carbon nanotubes and to a device therefor.

Carbon nanotubes are recently discovered, hollow graphite tubules having a diameter of generally several to several tens nanometers. One method for the preparation of carbon nanotubes is disclosed in Nature, 358, 220 (1992) by Ebbesen et al. In this method, a DC potential of about 18 V is applied between a 6 mm diameter graphite rod (anode) and a 9 mm diameter graphite rod (cathode) which are coaxially disposed in a reaction vessel maintained in an inert gas atmosphere. By positioning the two rods close to each other, an arc discharge occurs with the simultaneous deposition of a carbonaceous material containing carbon nanotubes on the cathode rod. Since the anode rod is consumed as the arc discharge proceeds, the anode (or cathode) is occasionally displaced to maintain a constant gap.

The known method has a problem because it is not possible to continuously produce carbon nanotubes in a large amount. Further, since carbonaceous deposits are gradually accumulated on the cathode, it is difficult to maintain the optimum gap between the anode and the cathode and, thus, to maintain the optimum conditions for the production of carbon nanotubes.

The present invention has been made from a consideration of these problems of the known method of producing carbon nanotubes.

In accordance with one aspect of the present invention there is provided a method of producing carbon nanotubes, comprising the steps of:

(a) successively positioning an axially extending rod-like carbonaceous anode having a tip end surface and a cathode having a cathode surface having an area greater than that of said tip end surface in an atmosphere of an inert gas such that said tip end surface successively faces on different portions of said cathode surface while keeping the distance therebetween substantially unchanged and while keeping the axis of said rod-like anode oriented in the direction normal to each of said portions of said cathode surface;
(b) impressing a direct current voltage between said anode and said cathode to cause an arc discharge to occur between said tip end surface of said anode and each of said portions of said cathode surface and to permit carbonaceous deposits containing carbon nanotubes to accumulate on each of said portions; and
(c) scraping said carbonaceous deposits from each of said portions.

In another aspect, the present invention provides a device for producing carbon nanotubes, comprising:

an airtight chamber;
an axially extending rod-like carbonaceous anode having a tip end surface;
a cathode having a cathode surface having an area larger than that of said tip end surface of said anode;
means for feeding an inert gas to said chamber;
means for supporting said cathode such that said cathode surface thereof is positioned within said chamber;
means for supporting said anode such that said tip end surface is positioned within said chamber and faces on said cathode surface with the axis of said rod-like anode being oriented in the direction normal to said cathode surface;
first means for controlling the distance between said tip end surface and said cathode surface;
second means for successively positioning said anode and said cathode such that said tip end surface successively faces on different portions of said cathode surface;
means for impressing a direct current voltage between said anode and said cathode so as to cause an arc discharge to occur between said tip end surface of said anode and each of said portions of said cathode surface and to permit carbonaceous deposits containing carbon nanotubes to accumulate on each of said portions of said cathode surface; and
means for scraping the carbonaceous deposits from each of said portions of said cathode surface.

The invention will now be described further with reference to the accompanying drawings, in which:

Fig. 1 is a schematic illustration of a preferred embodiment of a device for the production of carbon nanotubes according to the present invention;
Fig. 2 is a schematic illustration of the arrangement of an anode, a cathode and a blade in the device of Fig. 1; and
Figs. 3 and 4 are schematic illustrations, similar to Fig. 2, showing other arrangements.

Referring to Figs. 1 and 2, designated as 1 is an airtight chamber in which an arc discharge is to be carried out. An axially extending rod-like anode 6 having a tip end surface 6a is horizontally disposed within the chamber 1. The anode 6 is formed of a carbonaceous material such as carbon, graphite or metal-containing graphite. The metal of the metal-containing graphite may be, for example, copper, iron or cobalt. The diameter of the anode 6 is generally 5-30 mm, preferably 8-15 mm. The anode 6 is supported by a holder (not shown) which in turn is connected to a position adjuster 7. The position adjuster 7 is operated manually or automatically to axially displace the anode 6 so as to position the tip end surface 6a thereof at a desired location. The holder is electrically connected to a positive pole of a direct current source 9 through an ammeter 12.

A cathode 2 having a cathode surface 2a is also disposed within the chamber 1 such that the cathode surface 2a is oriented normal to the axis of the rod-like anode 6. It is important that the area of the cathode surface 2a is larger than that of the tip end surface 6a of the anode 6. Preferably, the cathode surface 2a is at least 4 times as large as the tip end surface 6a. The cathode 2, which is formed of a heat-resisting conductive material such as a metal, e.g. copper, or a carbonaceous material such as carbon, graphite or metal-containing graphite, is in the form of a cylinder whose axis is oriented in parallel with the axis of the anode 6 but is offset from the axis of anode 6.

Designated as 3 is a support member fixed within the chamber 1 and having a hole in which the cylindrical cathode 2 is rotatably received such that the axis of the cathode 2 serves as the center of rotation thereof. The support member 3 is made of a conductive member and is electrically contacted with the cathode 2. The support member 3 is coupled to a negative pole of the direct current source 9.

The cylindrical cathode 2 has an end surface which is opposite to the cathode surface 2a and to which a coaxial, electrically insulating shaft 4 is secured for rotation with the cathode 2. The shaft 4 extends out of the chamber 1 and is connected to a driving mechanism 5 including an electric motor for rotating the shaft 4.

As a result of the above construction, by mounting the rod-like anode 6 on the holder, the tip end surface 6a faces in the direction of the cathode surface 2a of the cathode 2. By operating the position adjuster 7, the gap between the tip end surface 6a and the cathode surface 2a is adjustable at will. By operating the driving mechanism 5, the cathode 2 is rotated so that the anode surface 6a can face on different portions of the cathode surface 2a.

Designated as 11 is a source of an inert gas such as helium, argon or nitrogen for feeding the inert gas of a controlled pressure to the chamber 1. A pressure gauge 13 is connected to the chamber 1 for measuring the inside pressure of the chamber 1. Designated as 10 is an evacuator.

A scraping member 8 is disposed within the chamber 1 for scraping carbonaceous deposits formed on the surface of the cathode surface 2a. The scraping member 8 in the illustrated embodiment includes a blade whose edge 8a is maintained in contact with the cathode surface 2a.

The structure of the cathode may be changed in various ways. In the alternative embodiment shown in Fig. 3, the peripheral surface of the cylindrical cathode 2 provides the cathode surface 2a. The rotational axis of the cathode 2 is oriented normal to that of the anode 6. By rotation of the cathode 2, the tip end surface 6a of the anode successively face on different portions of the cathode surface 2a. In the further embodiment shown in Fig. 4, the cathode 2 is reciprocally displaced in the direction normal to the axis of the anode 6 and the cathode surface 2a extends in the direction parallel with the reciprocating direction. If desired, the displacement of the tip end surface 6a of the anode 6 relative to the cathode surface 2a in the foregoing embodiments may be performed manually.

While, in the foregoing embodiments, the cathode 2 is movably constructed, it is possible to stationarily fix the cathode 2 within the chamber 1 with the anode 6 being arranged to be moved not only in the axial direction but also in parallel with the cathode surface 2a. However, since it is also necessary to move the scraping member 8 and parts associated with the anode 6, the structure becomes more complicated as compared with the foregoing illustrated embodiments.

A preferred method of producing carbon nanotubes according to the present invention using the device of Fig. 1 will be now described.

In starting up, the anode 6 is displaced to bring the tip end surface 6a into contact with the cathode surface 2a. The evacuator 10 is operated to reduce the pressure within the chamber 1 to 0.1-760 Torr, preferably 1-20 Torr and, then, the direct current voltage source 10 is connected to the anode 6 and the cathode 2 to heat them while continuing the evacuation. By this pretreatment, oxygen and moisture entrapped in respective parts within the chamber are withdrawn therefrom.

Helium gas is then fed from the source 11 to the chamber 1 and the helium gas pressure is maintained at 10 Torr to 2 atm, preferably at 500 Torr to 1 atm. Thereafter, the position adjuster 7 is operated to adjust the distance between the tip end surface 6a and the anode surface 2a to generally 0.1-5 mm, preferably 0.5-2 mm, while impressing the direct current voltage of generally 15-35 V, preferably 18-21 V therebetween, so that an arc discharge occurs with the simultaneous deposition of a carbonaceous material containing carbon nanotubes on that portion of the cathode surface 2a which is adjacent to the tip end surface 6a of the anode 6. The DC current in this case is controlled to 100-200 A.

While continuing the arc discharge, the driving mechanism 5 is continuously or intermittently operated to rotate the cathode 2 and to change the relative position between the tip end surface 6a and the cathode surface 2a. The rotational speed may be such that the average running speed of the tip end surface 6a relative to the cathode surface 2a ranges from 60 to 300 mm/minute. During the rotation of the cathode 2, the carbonaceous deposits are scraped by the blade 8 and collected. The distance between the cathode surface 2a and the tip end surface 6a is also controlled in the above range since the anode is consumed as the arc discharge proceeds.

The following example will further illustrate the present invention.

Example

Carbon nanotubes were produced using the device

shown in Fig. 1. A graphite rod having a diameter of 15 mm was used as the anode 6 while a cylindrical graphite rod having a diameter of 65 mm was used as the cathode 2. The anode 6 and the cathode 2 were disposed within the chamber so that the axis of the anode 6 was spaced apart by a distance of 25 mm from the axis of the cathode 2. After locating the anode 6 in abutting engagement with the cathode 2, the chamber 1 was evacuated. While maintaining the chamber 1 at a pressure of about 1 Torr, a DC current was allowed to flow through the anode 6 and cathode 2 for 3 hours to heat the anode 6 and cathode 2. Then, helium gas was continuously fed to the chamber 1 at a rate of 10 liters per minute under a pressure of 1 atm. The anode 6 was slightly retracted to form a gap of 1 mm between the tip end surface 6a and the cathode surface 2a, so that arc discharge occurred. While maintaining the electrical current at 100 A and the gap between the anode and the cathode at 1 mm, the cathode 2 was rotated at a rate of about 10 revolutions per minute. Thus, the carbonaceous material deposits formed on the cathode surface 2a were continuously scraped with the blade 8 and collected in the bottom of the chamber 1. The yield of the carbonaceous material was about 1 g per hour. The SEM photograph revealed that carbon nanotubes were contained in the carbonaceous material.

## Claims

1. A method of producing carbon nanotubes, comprising the steps of:

   (a) successively positioning an axially extending rod-like carbonaceous anode having a tip end surface and a cathode having a cathode surface having an area greater than that of said tip end surface in an atmosphere of an inert gas such that said tip end surface successively faces on different portions of said cathode surface while keeping the distance therebetween substantially unchanged and while keeping the axis of said rod-like anode oriented in the direction normal to each of said portions of said cathode surface;
   (b) impressing a direct current voltage between said anode and said cathode to cause an arc discharge to occur between said tip end surface of said anode and each of said portions of said cathode surface and to permit carbonaceous deposits containing carbon nanotubes to accumulate on each of said portions; and
   (c) scraping said carbonaceous deposits from each of said portions.

2. A method as claimed in claim 1, wherein step (a) is performed continuously.

3. A method as claimed in claim 1, wherein step (a) is performed intermittently.

4. A method as claimed in any preceding claim, wherein said cathode surface has an area at least 4 times as large as that of said tip end surface of said anode.

5. A device for producing carbon nanotubes, comprising:

   an airtight chamber;
   an axially extending rod-like carbonaceous anode having a tip end surface;
   a cathode having a cathode surface having an area larger than that of said tip end surface of said anode;
   means for feeding an inert gas to said chamber;
   means for supporting said cathode such that said cathode surface thereof is positioned within said chamber;
   means for supporting said anode such that said tip end surface is positioned within said chamber and faces on said cathode surface with the axis of said rod-like anode being oriented in the direction normal to said cathode surface;
   first means for controlling the distance between said tip end surface and said cathode surface;
   second means for successively positioning said anode and said cathode such that said tip end surface successively faces on different portions of said cathode surface;
   means for impressing a direct current voltage between said anode and said cathode so as to cause an arc discharge to occur between said tip end surface of said anode and each of said portions of said cathode surface and to permit carbonaceous deposits containing carbon nanotubes to accumulate on each of said portions of said cathode surface; and
   means for scraping the carbonaceous deposits from each of said portions of said cathode surface.

6. A device as claimed in claim 5,

   wherein said cathode includes an electrically conductive columnar body having a circular cross-section, said columnar body having one end providing said cathode surface and the other end fixedly connected to a coaxially extending, electrically insulating shaft,
   wherein said means for supporting said cathode includes a support member fixed within said chamber and having a hole in which said columnar body of said cathode is rotatably received such that the axis of said columnar body serves as the center of rotation of said columnar body,

wherein said second means includes drive means connected to said shaft for rotating said shaft about the axis thereof,
wherein said means for supporting said anode includes a holder holding said anode such that the axis of said anode is oriented in parallel with the axis of said columnar body but is offset from the axis of said columnar body,
wherein said first means includes drive means connected to said holder for shifting said holder in the direction parallel with the axis of said anode, and
wherein said scraping means includes a blade fixedly disposed within said chamber such that said blade is in sliding contact with each of said portions of said cathode surface upon rotation of said columnar body.

7. A device as claimed in claim 6, wherein said support member is formed of an electrically conductive material and is maintained in electrical contact with said columnar body, wherein said holder is formed of an electrically conductive material and is maintained in electrical contact with said anode, and wherein said means for impressing a direct current voltage includes lead wires extending between a DC source and said holder and between said DC source and said support member.

**Patentansprüche**

1. Eine Methode zur Herstellung von Kohlenstoffnanoröhren, die die folgenden Schritte umfaßt:

   (a) Aufeinanderfolgendes Positionieren einer axial ausgerichteten stabartigen kohlehaltigen Anode mit einer spitzenförmigen Abschlußfläche und einer Kathode mit einer Kathodenfläche, die größer ist als die spitzenförmige Abschlußfläche, in einer Schutzgasatmosphäre, so daß die spitzenförmige Abschlußfläche nacheinander verschiedenen Abschnitten der Kathodenfläche gegenüberliegt, während der Abstand zwischen beiden im wesentlichen konstant und die Achse der stabartigen Anode normal ausgerichtet zu den einzelnen Abschnitten der Kathodenfläche bleibt;

   (b) Anlegen einer Gleichspannung zwischen der Anode und der Kathode, so daß es zwischen der spitzenförmigen Abschlußfläche der Anode und den einzelnen Abschnitten der Kathodenfläche zu einer Lichtbogenentladung kommt und sich auf allen Abschnitten kohlehaltige Ablagerungen bilden können, die Kohlenstoffnanoröhren enthalten;

   (c) Abschaben der kohlehaltigen Ablagerungen von den einzelnen Abschnitten.

2. Eine Methode gemäß Anspruch 1, bei der Schritt (a) ständig abläuft.

3. Eine Methode gemäß Anspruch 1, bei der Schritt (a) diskontinuierlich abläuft.

4. Eine Methode gemäß einem der vorstehenden Ansprüche, bei der die Kathodenfläche wenigstens viermal größer ist als die spitzenförmige Abschlußfläche der Anode.

5. Eine Vorrichtung zur Herstellung von Kohlenstoffnanoröhren, die folgendes umfaßt:

   eine luftdichte Kammer;
   eine axial ausgerichtete stabartige kohlehaltige Anode mit spitzenförmiger Abschlußfläche;
   eine Kathode mit einer Kathodenfläche, die größer ist als die spitzenförmige Abschlußfläche der Anode;
   Vorrichtung zur Einleitung des Schutzgases in die Kammer;
   Vorrichtung für die Aufnahme der Kathode, die gewährleistet, daß sich die Kathodenfläche in der Kammer befindet;
   Vorrichtung für die Aufnahme der Anode, die gewährleistet, daß sich die spitzenförmige Abschlußfläche in der Kammer und gegenüber der Kathodenfläche befindet und die Achse der stabartigen Anode normal zur Kathodenoberfläche ausgerichtet ist;
   eine erste Einrichtung für die Regelung des Abstands zwischen der spitzenförmigen Abschlußfläche und der Kathodenfläche;
   eine zweite Einrichtung für die aufeinanderfolgende Positionierung der Anode und der Kathode, so daß die spitzenförmige Abschlußfläche verschiedenen Abschnitten der Kathodenfläche gegenüberliegt;
   eine Einrichtung für das Anlegen einer Gleichspannung zwischen Anode und Kathode, so daß es zwischen der spitzenförmigen Abschlußfläche der Anode und den einzelnen Abschnitten der Kathodenfläche zu einer Lichtbogenentladung kommt und sich auf den einzelnen Abschnitten der Kathodenfläche kohlehaltige Ablagerungen mit Kohlenstoffnanoröhren bilden können;
   eine Vorrichtung für das Abschaben der kohlehaltigen Ablagerungen von den einzelnen Abschnitten der Kathodenfläche.

6. Eine Vorrichtung gemäß Anspruch 5, wobei die Kathode einen elektrisch leitenden zylindrischen Körper mit einem kreisförmigen Querschnitt enthält,

das eine Ende des zylindrischen Körpers die Kathodenfläche liefert und das andere Ende fest mit einer koaxialen elektrisch isolierenden Welle verbunden ist,

wobei die Vorrichtung für die Aufnahme der Kathode eine Aufnahmeeinrichtung umfaßt, die in der Kammer angebracht und mit einer Öffnung versehen ist, in der der zylindrische Körper der Kathode drehbar gelagert ist, so daß die Achse des zylindrischen Körpers den Drehpunkt des zylindrischen Körpers darstellt,
wobei die zweite Vorrichtung eine mit der Welle verbundene Antriebsvorrichtung für das Drehen der Welle um die Achse umfaßt,
wobei die Vorrichtung für die Aufnahme der Anode eine Halterung für die Anode umfaßt, die gewährleistet, daß die Achse der Anode parallel zur Achse des zylindrischen Körpers ausgerichtet, jedoch versetzt zur Achse des zylindrischen Körpers ist,
wobei die erste Vorrichtung eine mit der Halterung verbundene Antriebsvorrichtung umfaßt, die die Halterung parallel zur Achse der Anode verschiebt, und
wobei die Abstreifvorrichtung eine fest in der Kammer angebrachte Klinge beinhaltet, die beim Drehen des zylindrischen Körpers in Gleitkontakt mit den einzelnen Abschnitten der Kathodenoberfläche ist.

7. Eine Vorrichtung gemäß Anspruch 6, wobei das Aufnahmeelement aus einem elektrisch leitfähigen Material besteht und elektrischen Kontakt mit dem zylindrischen Körper hat, wobei-die Halterung aus einem elektrisch leitfähigen Material besteht und elektrischen Kontakt mit der Anode hat und wobei die Vorrichtung für das Anlegen einer Gleichspannung Leitungsdrähte zwischen der GS-Quelle und der Halterung und zwischen der GS-Quelle und dem Aufnahmeelement besitzt.

## Revendications

1. Procédé de production de nanotubes de carbone, comprenant les étapes consistant à:

(a) positionner successivement une anode carbonée en forme de tige, s'étendant axialement, présentant une surface d'extrémité en pointe et une cathode présentant une surface cathodique dont l'aire est supérieure à celle de ladite surface d'extrémité en pointe dans une atmosphère de gaz inerte, de telle sorte que ladite surface d'extrémité en pointe fasse successivement face à différentes portions de ladite surface cathodique tout en conservant la distance entre elles sensiblement inchangée, tout en conservant l'axe de ladite anode en forme de tige orienté dans la direction normale à chacune desdites portions de ladite surface cathodique;
(b) appliquer une tension continue entre ladite anode et ladite cathode pour provoquer une décharge d'arc entre ladite surface d'extrémité en pointe de ladite anode et chacune desdites portions de ladite surface cathodique et pour permettre l'accumulation de dépôts carbonés contenant des nanotubes de carbone sur chacune desdites portions; et
(c) gratter lesdits dépôts carbonés depuis chacune desdites portions.

2. Procédé selon la revendication 1, dans lequel l'étape (a) est réalisée de façon continue.

3. Procédé selon la revendication 1, dans lequel l'étape (a) est réalisée de façon intermittente.

4. Procédé selon l'une quelconque des revendications précédentes, dans lequel ladite surface cathodique présente une aire au moins 4 fois plus importante que celle de ladite surface d'extrémité en pointe de ladite anode.

5. Dispositif pour produire des nanotubes de carbone, comprenant:

une chambre étanche à l'air;
une anode carbonée en forme de tige, s'étendant axialement, présentant une surface d'extrémité en pointe;
une cathode présentant une surface cathodique dont l'aire est supérieure à celle de ladite surface d'extrémité en pointe de ladite anode;
un moyen pour alimenter ladite chambre en un gaz inerte;
un moyen pour supporter ladite cathode de sorte que ladite surface cathodique de celle-ci soit positionnée à l'intérieur de ladite chambre;
un moyen pour supporter ladite anode de sorte que ladite surface d'extrémité en pointe soit positionnée à l'intérieur de ladite chambre et fasse face à ladite surface cathodique, l'axe de ladite anode en forme de tige étant orienté dans la direction normale à ladite surface cathodique;
un premier moyen pour commander la distance entre ladite surface d'extrémité en pointe et ladite surface cathodique;
un deuxième moyen pour positionner successivement ladite anode et ladite cathode de telle sorte que ladite surface d'extrémité en pointe fasse successivement face à différentes portions de ladite surface cathodique;
un moyen pour appliquer une tension continue

entre ladite anode et ladite cathode pour provoquer une décharge d'arc entre ladite surface d'extrémité en pointe de ladite anode et chacune desdites portions de ladite surface cathodique et pour permettre l'accumulation de dépôts carbonés contenant des nanotubes de carbone sur chacune desdites portions de ladite surface cathodique; et

un moyen pour gratter lesdits dépôts carbonés depuis chacune desdites portions de ladite surface cathodique.

6. Dispositif selon la revendication 5,

dans lequel ladite cathode comporte un corps en colonne à section transversale circulaire conducteur de l'électricité, ledit corps en colonne présentant une extrémité fournissant ladite surface cathodique et l'autre extrémité connectée de manière fixe à un arbre électriquement isolant s'étendant coaxialement,

dans lequel ledit moyen pour supporter ladite cathode comporte un organe de support fixé à l'intérieur de ladite chambre et présentant un trou dans lequel est reçu en rotation ledit corps en colonne de ladite cathode, de telle sorte que l'axe dudit corps en colonne serve de centre de rotation dudit corps en colonne,

dans lequel ledit deuxième moyen comporte un moyen d'entraînement connecté audit arbre en vue d'entraîner en rotation ledit arbre autour dudit axe de celui-ci,

dans lequel ledit moyen pour supporter ladite anode comporte un élément porteur portant ladite anode de telle sorte que l'axe de ladite anode soit orienté parallèlement à l'axe dudit corps en colonne, en étant toutefois décalé par rapport à l'axe dudit corps en colonne,

dans lequel ledit premier moyen comporte un moyen d'entraînement connecté audit élément porteur pour déplacer ledit élément porteur dans la direction parallèle à l'axe de ladite anode, et

dans lequel ledit moyen de grattage comporte une lame disposée de manière fixe à l'intérieur de ladite chambre de telle sorte que ladite lame soit en contact glissant avec chacune desdites portions de ladite surface cathodique lors de la rotation dudit corps en colonne.

7. Dispositif selon la revendication 6, dans lequel ledit organe de support est formé d'un matériau conducteur de l'électricité et est maintenu en contact électrique avec ledit corps en colonne, dans lequel ledit élément porteur est formé d'un matériau conducteur de l'électricité et est maintenu en contact électrique avec ladite anode, et dans lequel ledit moyen pour appliquer une tension continue comporte des fils conducteurs s'étendant entre une source continue et ledit élément porteur et entre ladite source continue et ledit organe de support.

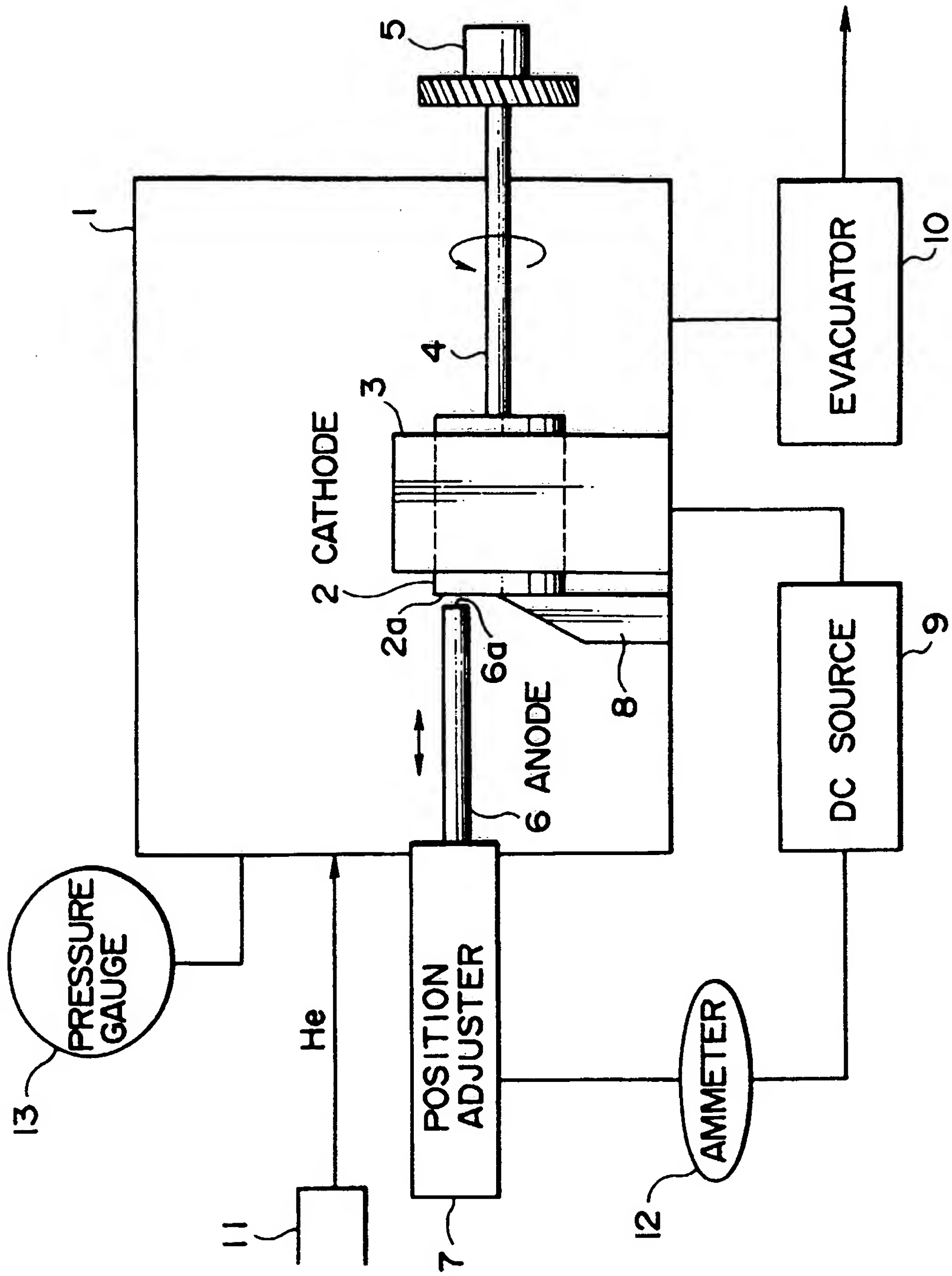
PRESSURE GAUGE
13
He
11
POSITION ADJUSTER
7
AMMETER
12
DC SOURCE
9
EVACUATOR
10
1
2 CATHODE
2a
6a
6 ANODE
3
4
5
8

FIG. 1

FIG. 2

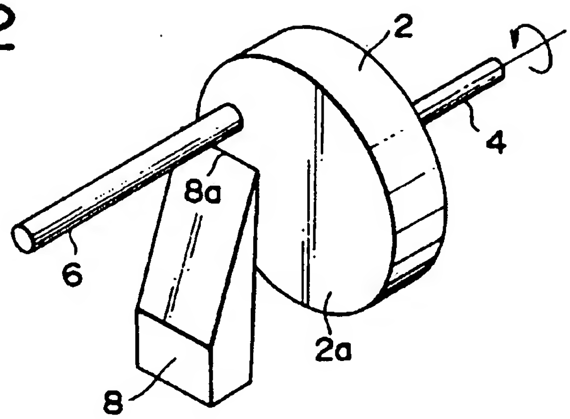


FIG. 3

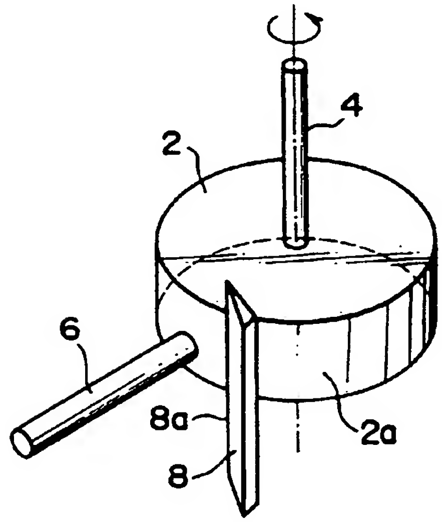


FIG. 4